

THE ANALYST.

NOVEMBER, 1894.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE usual Monthly Meeting of this Society was held on October 3rd at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Otto Hehner took the chair.

The minutes of the last meeting were read and confirmed.

The following gentlemen were proposed for election as members: E. A. Hancock, F.C.S., Government Laboratory, St. Kitts, West Indies (by the Council); Thomas Alex. Pooley, B.Sc. (London), F.I.C., 34, Old Broad Street, E.C.; John Clough Thresh, D.Sc. (London), M.B., B.Sc. (Victoria), D.P.H., Chelmsford, Essex; Robert Waterhouse, 101, Leadenhall Street, E.C.; William G. Wagner, 101, Leadenhall Street, E.C.

Mr. Bevan then read the following paper:

THE LOSS OF TOTAL SOLIDS IN MILK ON KEEPING.

SOME time ago a number of samples of milk arrived at my laboratory late on Saturday morning. The specific gravity of each was taken, and the fat estimated by the Leffmann-Beam process. Such as were suspected of being adulterated were weighed out into platinum dishes, and placed in desiccators until the following Monday morning, when they were evaporated to dryness in the usual way. My assistant, Mr. Merriman, called my attention to the fact that the total solids found in every case differed by about 1.0 per cent. from that calculated by the Richmond scale. As a rule, we find the concordance between the calculated and observed total solids to be very close, and we were at a loss to account for the discrepancy. The total solids were repeated on fresh portions of the milks, when they were found to correspond with the calculated quantities. Further analysis showed the milks to be perfectly normal as regards the relationship between gravity, fat, and total solids.

The discrepancy was so marked in every instance that we were forced to the conclusion that a loss of total solids had taken place on standing, and we immediately instituted a number of experiments with a view to showing if this really were the case. The following are the results obtained:

TABLE A.

	Total Solids.	Loss in Total Solids.
Evaporated immediately standing in open dish	11.73	
“ after 24 hours	10.79	0.94
“ after 48 hours	10.38	1.35
“ after 120 hours	9.42	2.31

A large number of experiments were made showing similar results.

I communicated the results of my experiments to Mr. Richmond, who informed me that Dr. Veith had observed the same thing, but in every case his loss in total solids was much less than mine. Notwithstanding that his results are published in *THE ANALYST* for 1882, the phenomenon seems to have been unknown to several chemists with whom I discussed the matter; nor have I been able to discover any notice of the fact in any work treating of milk analysis, beyond the following statement in Leffmann and Beam's book on "The Analysis of Milk and Milk Products": "Richmond has pointed out that if the evaporation be slow, some decomposition occurs, and the residue will be brown, but that if the larger portion of the water be evaporated quickly, a white residue is obtained."

If such a loss as 0.94 occurs in twenty-four hours, it is certain that an appreciable loss may arise in a very few hours, especially in warm weather. The importance of the matter must be my excuse for reading this paper to-night.

In the experiments I have already recorded, the milks were weighed out in the fresh state and evaporated at certain intervals without neutralizing, so that the loss includes everything volatile at the time of evaporation. If immediately before evaporation the milk is neutralized with decinormal soda the loss is not so great, probably due to the fact that the lactic or other acid formed is completely retained by the soda. Lactic acid is stated in text-books to be non-volatile at 100°, and Dr. Bell in "The Chemistry of Foods," Part II., says (p. 15), "and as the acid is not volatile, its weight is correctly indicated on drying the milk."

Actual experiment in my laboratory shows that lactic acid is volatile. To 5 grammes of fresh milk I added 0.1445 pure lactic acid. The total solids obtained amounted to 0.6610 grammes, as against 0.5784 in the original milk. This corresponds to a loss of 42.8 per cent. of the lactic acid. By merely evaporating a weak solution of lactic acid in a platinum dish at 100° a considerable loss is sustained. I have, moreover, made a number of experiments showing the difference in the solids-not-fat in sour milk between neutralizing and not neutralizing:

TABLE B.

	Original Milk.	Kept 48 Hours.	Kept 96 Hours.
Total solids neutralized	13.30	13.25	13.00
" " not neutralized	13.30	13.12	12.70
Difference	0.03	0.13	0.30
Total acidity reckoned as lactic acid ...	0.23	0.93	1.05

The samples were preserved in well-corked bottles.

In the following table (C) will be found the results obtained after neutralizing, together with the acidity in the case of milks kept in open dishes:

TABLE C.

	Loss in total Solids. Neutralized	Total Acid reckoned as Lactic.
Evaporated after standing in open dish } for 24 hours	0.30	0.220
Evaporated after 48 hours	0.65	0.630
" after 96 hours	0.88	0.594

It is important to notice that after standing ninety-six hours the loss in total solids amounts to 0·88 per cent., with a total acidity of 0·594 per cent. On comparing these numbers with those obtained with milks that have been kept in well-corked bottles (table B), it will be found that there is absolutely no connection between the loss and the percentage of acid formed. Moreover, it is remarkable that the acidity is greater in the milk kept for forty-eight hours than it is in that kept for ninety-six. It may be argued that a mistake has possibly been made either in the estimation of the total solids or in the acid; but numerous examples of similar "discrepancies" convince me at least that the differences are due to slight unknown variations in the conditions under which the samples have been kept. The following table shows the losses sustained by keeping milk under varying conditions, together with the amounts of total acid:

TABLE D.

Total Acidity reckoned as Lactic Acid.	Loss in total Solids (neutralized).	Conditions of keeping.	Composition of Original Milk.	
			Total Solids.	Fat.
1. 0·226	0·30	24 hours in open dish	12·38	3·50
2. 0·630	0·65	48 " "	"	"
3. 0·594	0·88	96 " "	"	"
4. 0·700	1·36	120 " "	"	"
5. 0·860	2·80	120 hours in open dish + 2% Na_2CO_3 ...	"	"
6. 0·50	1·16	120 hours in open dish + 2% salicylic acid ...	"	"
7. 0·774	2·17	120 hours in narrow beaker	"	"
8. 0·709	1·35	120 hours in flask with current of air passing ...	"	"
9. 1·04	0·74	51 days in corked bottle	9·20	0·29
10. 0·93	1·22	67 " "	12·00	3·28
11. 1·61	0·35	71 " "	11·50	3·54
12. 0·96	1·05	72 " "	11·51	3·18

The important point to be noticed is the disproportion between the total acidity and the loss in total solids.

If, then, there are such extraordinary differences in the ratio of the loss in total solids to the amount of total acidity (and the same thing would doubtless apply to the alcohol, ammonia, and acetic acid formed), one is inclined to ask what value, if any, is to be attached to a system of estimating these bodies in milks decomposed beyond recognition, and endeavouring to arrive at an opinion as to the original condition of the sample. Although reference has already been made in this journal, I cannot do better than repeat in this connection the questions put to Mr. Bannister and the answers he gave before the Select Committee:

1669. And in order to allow for decomposition that has taken place you have a method of calculation?—We see what the change consists of, and work it back again into solids-not-fat.

1678. May not some of these milks fail to give the same results?—They will not give the same results. If you take two samples of reference milk the results obtained will not be exactly the same, because they have been kept under different conditions;

but we take out the different substances that have been formed, and work them back again into solids-not-fat, so that if there is a quantity of alcohol formed in the one, and in the other there is none, the alcohol would be worked back again into solids-not-fat.

1682. You do not think that any of the conclusions which you arrive at by that process would be such as would cause you to give a decision contrary to that arrived at by public analysts?—Practically, if the former analyses have been correctly made, they would agree with ours.

1686. And when you detect preservatives, do you adopt a different procedure in coming to your final decision?—No; we take the results of the analysis, the quantity of alcohol formed, of ammonia, of acetic acid, and so on, and turn it back again into solids-not-fat.

1689. What are the same?—The two systems, if you take the time allowance, and take our present method of examination; only no doubt our present system is the better system from a chemical point of view, because we take the determination of the different substances in the milk, and the changes that have taken place, and work them back again into solids-not-fat, so that it does not depend upon a time allowance.

With regard to the determination of alcohol: As a rule, the samples referred to Somerset House are twenty-eight days old; now the old time allowance, which, as Mr. Bannister has stated, is practically the same as the new and, from a chemical point of view, better system, is, according to Dr. Bell, 0.48 per cent. I think it may be taken for granted that in no case does the weight of the sample exceed 6 ounces. Six ounces is equal to 168 grammes; 0.48 per cent. on 168 grammes is 0.806 gramme, which represents the total actual loss, and includes, of course, alcohol and carbonic acid.

Even if the whole of this loss of 0.806 gramme were alcohol, its accurate determination would be an exceedingly difficult matter. But when we consider that the total solids have also to be determined, presumably in duplicate, the difficulty is increased. But of course a considerable proportion of the loss is represented by carbonic acid, so that the amount of alcohol that can possibly be present would be very small. A number of experiments are in progress which I hope will throw light on the relationship between the loss in solids and the carbonic acid evolved.

DISCUSSION.

Mr. Hehner said that Mr. Bevan had given them a series of very interesting observations on the subject of the alterations which occurred in milk during keeping. Though this subject had been often discussed on previous occasions in that room, yet it was desirable that it should be approached by a new mind and from a different point of view. Mr. Bevan's observations when further expanded could not fail to be of considerable utility.

Mr. Richmond said that in a series of experiments which he had made some years since, he had found that lactic acid, especially in concentrated solution, was distinctly volatile when distilled with water. There was not nearly the same loss with the commercial syrupy acid, because it contained a considerable proportion of

anhydride, and it had to be boiled for some time before the anhydride was converted into the acid. With regard to the loss of total solids on keeping milk, taking Mr. Bevan's figures, it appeared, roughly speaking, the greater amount of acid formed, the less was the loss—a fact which his own experience confirmed. In some samples there was a large amount of acid formed and a moderate loss of total solids, but in one sample, which had been kept for six or seven weeks, though only a small amount of acid had been formed, there was a loss of 5 or 6 per cent. in the total solids. Mr. Stokes had published results of the same kind. In Dr. Bell's book it is stated that when there is an excessive amount of acid formed, a greater amount of decomposition was indicated, and presumably a greater loss of total solids. During the last three or four years there had appeared in the journal of the Chemical Society a series of papers by Dr. Percy Frankland on the fermentation of sugar with pure cultivations of different organisms; and it had been found that there was a great difference in the nature of the products according as the fermentation was conducted in an open or a closed vessel. In the former case the products were generally alcohol, acetic acid, and frequently other acids (succinic); in the latter, alcohol, acetic acid, and *formic acid*, while much hydrogen and carbon dioxide were evolved. The ethacetic fermentation was found to be very general, and the quantitative yields of alcohol, etc., varied within fairly wide limits. Dr. Frankland's researches indicate very strongly that in keeping milk in a bottle there was likely to be considerable difference in the products of decomposition according as the bottle was tightly corked or the reverse. The products of the decomposition of milk were so varied that it was impossible to fix any time allowance. The estimation and calculation back of the alcohol and other bodies formed seemed to be of doubtful accuracy. He had found in the case of koumiss, which was milk fermented by the action of various organisms under constant conditions, that it was possible to find an approximately constant factor, though hardly good enough for analytical purposes; but it is extremely doubtful if this can be done when milk is subjected to the action of all kinds of organisms. In reply to Mr. Bevan, Mr. Richmond said that koumiss was not fermented by a pure culture of bacteria, but by a special cultivation in which several (at least three) species of organisms were present.

Mr. Allen said that at the present time he had a series of experiments in progress with a view of learning more about the rate of change of milk by keeping. A number of tubes were taken, and 10 grammes weight of a sample of new milk placed in each. The tubes were then hermetically sealed, and preserved at the ordinary temperature. Every week one of the tubes was opened, the contents transferred to a dish, and the liquid evaporated to dryness at 100° C. It might have been better to neutralize the milk previously to evaporation, but as a matter of fact this had not been done. At the same time that this series of tubes were sealed, two other series were prepared by adding to the same milk one-fourth of its measure of tap-water in the one case, and canal-water in the other. It was thought that these would represent pure and impure waters respectively. The diluted milk was then sealed up in quantities of 10 grammes, as in the case of the pure milk, and one tube of each series opened every week. The experiments, which were conducted at

his request by Mr. Haywood Court, were still in progress, but the following general results might be mentioned. The solids in the case of pure milk fell in fifty-two days from 12.58 to 11.21 per cent., being a loss of 1.37 per cent. The diluted milks lost in the same time 1.44 and 1.46 per cent. respectively. These experiments do not show the varying rate of change which has been observed in other cases to be produced by dilution or the addition of impure water. On the other hand, they exhibit a marked disagreement with the change alleged by the Somerset House chemists to occur in milk by keeping. Had their notorious time allowance for change been correct, the loss of solids in fifty-two days would have been 0.68 per cent., while the amount actually lost was exactly twice as much.

The variable rate of change in milk by keeping was discussed in an able paper read before the Society some years since by Mr. Stokes (*ANALYST*, xii., 226), and in the discussion numerous figures were given conclusively proving the utter worthlessness of the referees' time allowance for change. The Somerset House system of constant allowance was perhaps the most preposterous practice ever devised by persons occupying a responsible position, and it was pitiable to reflect that it had been adhered to for a long series of years, in spite of its palpable absurdity and its repeated impeachment by all those best qualified to judge. The referees' time allowance might well be permitted to sink into the obscurity it deserved, were it not that Mr. Bannister, in his evidence before the Committee on Food Products Adulteration, said that it gave substantially the same results as the system of compensation now employed, which latter he insisted was thoroughly trustworthy. He admitted that the presence of a preservative affected the rate of change, but said that the two processes gave substantially the same results. This would appear to mean that the present plan gave results agreeing with those of the time allowance, no matter whether a preservative had been added or not. This was tantamount to the proposition that things which are equal to the same thing are not equal to each other. As to the revised version of allowance-making, he would merely point out that its inventors had not ventured to present it for discussion before any competent tribunal or to publish the details in scientific journals; and as they were very easily satisfied in the case of the time allowance, it was probable that they were content with a minimum of accuracy in the results of their new system.

It must be admitted that the Somerset House Chemists had been placed in a very difficult position by having submitted to them samples of milk which every chemist knew were unfit for analysis. If, instead of pretending to accomplish impossibilities, the referees had eighteen years ago certified in intelligible terms their inability to form a definite opinion on the samples of milk submitted to them, the law would very quickly have been modified. In only four cases, he believed, in the course of eighteen years had the referees certified that samples of milk submitted to them were not in a fit condition for analysis, and these cases were comparatively recent.

The evidence given by Mr. Bannister was a revelation to public analysts in many respects, but not even his ignorance of the Society's limit for fat astonished them more than his statement that the words "we are unable to affirm," which have caused so much heart-burning, were intended by the referees to indicate to

the court that they could not express a definite opinion on the sample either one way or the other! It would be interesting to ascertain how many scores of failures of justice were directly attributable to the misleading or ambiguous wording of the referees' certificates, and how many real cases of non-confirmation of public analysts' certificates would remain after deducting the former class.

With regard to the preservation of milk samples, Mr. Allen said that a portion of every adulterated sample which passed through his hands was preserved by adding to it twice its weight of alcohol. In one case, the sample so preserved was analysed after several weeks by Mr. Hehner and Mr. Estcourt, and their results served to refute the certificate given at Somerset House, and prevent the failure of justice which would have resulted had the referees' certificate been accepted. But he was not satisfied with the use of alcohol as a preservative, and had tried several other substances, with very limited success. Bichromate of potassium had been advocated, and had been thoroughly tried in his laboratory by Mr. W. G. Wagner, but the very considerable quantity of it required made it objectionable; otherwise it had the advantage that the actual amount which had been used could be ascertained with precision, and due allowance made accordingly. Hydrofluoric acid, chloroform, and carbon disulphide had also been tried, with limited success. What was wanted, in his opinion, was a solid preserving agent which could be added to the milk at the time of purchase in the form of pellets of definite weight, such an addition being legalized. If this were done in every case, the referees would receive their portions of the sample in a practically unchanged condition, the chief bone of contention with Public Analysts would be buried, and the grave loss of caste and credit which Somerset House had suffered through its ill-advised action in the past might ultimately be forgotten.

Mr. Bodmer said that Mr. Stokes had made some experiments which showed the utter unreliability of any such thing as a time allowance, and this confirmed Mr. Bevan's statements. Formaldehyde was now being much used for preserving milk for analytical purposes, under the name of "Formalin."

Mr. Richmond said that he had been using formaldehyde for the last year and a half; 0.05 per cent. would keep milk for a month, and larger quantities almost indefinitely.

Dr. Adams said that he had also used formaldehyde, and had found that the addition of five or six drops was sufficient to preserve a sample of milk for a week. Some of the points referred to in Mr. Bevan's paper had been alluded to in his (the speaker's) paper on the use of ammonia in the analysis of sour milk. He (Dr. Adams) had come to the conclusion that the loss experienced was in some measure due to loss of water of crystallization. We might well believe that the milk sugar might be caramellized by the acids formed during the decomposition of milk, when these acids were concentrated during the drying of the solids.

Mr. Cassal said that he could not agree with the suggestions of Mr. Allen and Mr. Bodmer that an antiseptic should be added to the milk by the inspector when taking the sample. He entirely disapproved of inspectors having anything to do with samples beyond taking them. Any other course would open the door to errors

of all kinds. The addition of a preservative by an inspector would render it impossible to take action successfully against a vendor for using preservatives. The sole object of the suggestion to add a preservative was to enable the Somerset House chemists to make analyses which would enable them to give an opinion approaching to accuracy with respect to the composition of a milk when fresh. The Somerset House analysts should have plainly stated at the outset that it was an absolute impossibility to arrive at a knowledge of the composition of a milk when fresh from an analysis made of the milk when decomposed. After the position they had taken up, and actually defended, it was hardly to be expected that they would now publicly acknowledge themselves to be absolutely wrong by accepting such a proposition. That kind of thing was hardly characteristic of Government Departments. He (Mr. Cassal) had noticed similar, and, in fact, considerably greater, diminution in total solids on keeping milk; there was no regularity about it, and it was unscientific and absurd to suppose that there would be. The differences in the extent of the loss must depend upon the temperature at which the sample was kept, and on the nature of the organisms which had obtained access to it.

Mr. Northfield Yarrow said that he had also noticed a great loss in milk solids when they were not evaporated as rapidly as possible, and he found this out in much the same way as Mr. Bevan had done. Milks were sometimes received late in the day, and the total solids were then weighed out and put inside the oven of the water-bath at once. The total solids thus obtained were much below those of the same milks which were evaporated outside the bath immediately after weighing. Sometimes the loss was as much as 2 per cent. With regard to formaldehyde, the 40 per cent. solution had been in use in Mr. Stokes' laboratory for over a year for preserving reference samples, and had given great satisfaction. One sample, which nine months ago had had this preservative added, was even then in an apparently fresh condition, but it had lost 0.4 per cent. in the total solids and 0.2 per cent. in the fat. For various reasons very little reliance was placed on these figures, but some series of experiments were now being made which might do something to show the rate of decomposition in milks when this compound was added.

Mr. Allen said that they must always have referees, and although they might not always see eye to eye with the gentlemen who at present occupied the position, they ought certainly to give them the utmost assistance in their power. It would be impossible for them to get their samples of milk in an undecomposed state until the addition of a preservative at the time of purchase was legalized. The nature and method of addition need not be specified by Act of Parliament, but might well be left to the Board of Reference, the formation of which they advocated, and then duly confirmed by Order in Council.

Mr. Hehner said he quite agreed with Mr. Cassal that inspectors should not be allowed to tamper in any way with samples. It was essential that an unaltered sample should be handed to the referees. But he did not think that the law laid on the referees the duty of giving an opinion on decomposed samples. He knew, indeed, that the referees had occasionally declined to examine samples which in their opinion were unfit for analysis; so, clearly, they were not under legal com-

pulsion, and he thought that it was never intended that such should be the case. The analysis of decomposed milk-samples was a proceeding which the Somerset House chemists had taken entirely on themselves; and it was a pity they did not at the beginning refuse to analyse decomposed samples, or, having analysed them, had not clearly stated in their certificates that they could not come to any definite conclusion. Public Analysts would certainly never have ventured to have given opinions on samples five or six weeks old—a course which had been regularly pursued at Somerset House. If the referees had been what they ought to be, truly scientific advisers of the Justices, they would have made some suggestion similar to Mr. Allen's. The system of allowances had been nothing short of scandalous. The Society had discussed them on many occasions, and Mr. Bevan had helped to clear up the subject. Officials, perhaps, could not be expected, after persisting in making erroneous allowances for seventeen or eighteen years, to confess now that they had been wrong all the time. We did not know with absolute certainty what takes place in the best-known fermentations—such as, for example, the alcoholic fermentation. Given a certain quantity of sugar, we did not know exactly how much alcohol and how much carbon dioxide were produced under all possible circumstances. If even this well-known process could not be expressed by a simple formula, what could be said about such products as those of the decomposition of a complex heterogeneous mass of albuminoids and other bodies acted on by an unknown variety of possibly unknown organisms? It was all very well to say that a molecule of milk-sugar was simply converted into two molecules of lactic acid, but no organism works for nothing. In the decomposition which it effects it evidently derives something for its own benefit, and life cannot be carried on without destructive metabolism. There must always be a loss between the stage of sugar and that of lactic acid, and what this is was unknown. Surely the day had gone past for regarding fermentation reactions as having the simplicity of text-book equations. As to the quantity of alcohol produced, we could only approximately calculate the quantity produced back to sugar; and he would like to know what the acetic acid was to be calculated into. Where did the acetic acid come from? From the alcohol or from the albuminoids?—the one was a small molecule, the other a large one. If the acetic acid and the ammonia were to be calculated into albuminoids, we got back to the latter from two sources. It was utterly unjustifiable to pretend to be able to make corrections for decomposition, and to compare the, at best, approximate figures thus obtained with those derived from the analysis of samples when fresh. From a merely analytical point, he would like to know how small quantities of acetic acid could be accurately determined in such complex bodies as the products of decomposed milk. The suggestion made by Mr. Bannister, in his evidence before the Parliamentary Committee, that such corrections for decomposition did not in any way concern the Public Analyst, illustrated his inability to appreciate the difficult position of the Public Analyst, who clearly was deeply concerned in a proceeding which affected his reputation.

Mr. Bevan, in reply, said that he agreed that the suggestion of Mr. Allen as to the inspectors being empowered to add preservatives was one to be deprecated.

Some arrangement by which the sample should be at once submitted to the referees would appear, however, to be desirable.

Several members here pointed out that such a course would involve a large expenditure, to which Mr. Bevan replied that possibly, then, as there were relatively few adulterated milks referred to Somerset House, whenever samples were found by the Public Analyst to be adulterated, the duplicates might be at once sent to the referees, if so desired by the vendor, without waiting for decomposition to progress.

In conclusion, Mr. Bevan pointed out that in his experience, as well as in that of others, the present referees under-estimated the fat in milk, and over-estimated, to an even greater extent, the solids-not-fat.

Estimation of Fat in Cheese. Stefan Bondzynski. (*Zeit. für Analyt. Chemie*, 1894; *Zweites Heft*, pp. 186-189.)—This is the application to cheese analysis of the Werner-Schmidt method of estimating fat in milk. A weighed quantity of the finely-divided cheese is placed in the tube, and decomposed with 20 c.c. HCl of sp. gr. 1.1, containing about 19 per cent. HCl. On cautiously warming over wire gauze the melted fat rises to the surface. After cooling, 30 c.c. of ether are added, and the tube warmed very gently, until the HCl and ethereal solution of fat separate sharply. Centrifugal force helps this, but is not essential. After the volume of ether has been read off, 20 c.c. are pipetted into a weighed Erlenmeyer flask. From this the quantity of fat in the entire solution can be calculated. The advantages claimed for the method are that it is rapid, and easily carried out.

The following results of cheese analyses are given :

	I.	II.	III.	IV.	V.	VI.
(1)	31.52	32.37	32.58	2.53	2.60	0.96 per cent. fat.
(2)	31.54	32.45	32.61	2.55	2.65	0.98 „ „

The fat in Cheeses I. and III., estimated by extraction in a Soxhlet, gave 31.43 per cent. and 32.62 per cent. respectively. Cheeses IV. and V. were made from centrifugal-skimmed milk. Cheese VI. had had its fat partially removed for analytical purposes.

C. A. M.

Estimation of Essential Oils, especially in Cloves and Mace. W. Lenz. (*Zeit. für Analyt. Chemie*, 1894; *Zweites Heft*, pp. 193-200.)—The solubility of oil of cloves in a 50 per cent. solution of sodium salicylate suggested to the author an improvement on the processes used to estimate the essential oils in spices. From 10 to 20 grammes of the substance is introduced into a retort holding 200 c.c., and having its beak inclined upwards, and then bent downwards at right angles at the middle, and connected with a condenser. Sufficient water is added to form a thin paste, and steam is passed through until the liquid distilling is free from every trace of oil. In order to prevent frothing, he introduces 10 c.c. of pure olive oil into the retort before distillation. The distillate (about 500 c.c.) is nearly saturated with sodium chloride, and shaken with successive portions of ether in a separating funnel.

It is usually sufficient to do this three times. The ethereal solution, freed from water by digesting for three or four days with calcium chloride, is evaporated in a weighed flask at 30°, a current of dried air being meanwhile passed through. It is weighed at intervals of five minutes until the weight becomes constant, and the quantity of eugenol is then estimated by Thom's method.*

By substituting a 50 per cent. solution of sodium salicylate for water in the retort, the author found that considerably higher results could be obtained. In the case of cloves, an average of 19.45 per cent. of oil, containing 84.52 per cent. of eugenol, was obtained, as compared with 17.75 per cent. of oil, containing 79.44 per cent. of eugenol, in the water distillation.

With mace, the oil of which is nearly insoluble in sodium salicylate solution, the method yielded an average of 8.41 per cent. of oil, while distillation with water alone only yielded 6.73 per cent. That the increased quantity of oil is not due to the boiling-point of the water being raised above 100° by the sodium salicylate is proved by experiments in which calcium chloride and potassium acetate are substituted for sodium salicylate. In these cases the quantity of oil yielded is almost identical with that yielded by the distillation with water alone. The author suggests as the explanation the peculiar solvent action of the sodium salicylate on the plant tissues which enclose the essential oil.

C. A. M.

The Decomposition of Albuminoids by Alkaline Hydrates. Dr. Victor Vedrödi. (*Zeit. für Analyt. Chemie*, 1894; *Drittes Heft*, pp. 338-340.)—The author's experiments prove—

- (1) That albuminoid substances are decomposed by boiling with sodium hydrate, ammonia being produced.
- (2) That the decomposition is slow, and even after twelve hours' boiling is not complete.

This raises the question: To what extent in tobacco analysis does the quantity of ammonia formed under such circumstances affect the estimation of the nicotine?

C. A. M.

On Lard. Dr. Samuelson. (*Zeit. für Analyt. Chemie*, 1894; *Heft 2*, pp. 189-192.)—Experiments on qualitative tests for detecting adulteration in lard are described at length. The conclusion arrived at is that, since all the recommended qualitative reactions may give negative reactions with a lard known to be adulterated with vegetable oil, the iodine number remains, as heretofore, the only reliable test.

C. A. M.

Method for the Determination of the Freezing-Point of Fatty Acids. F. Wolfbauer. (Translated and abridged by S. S. Emery.) (*Journal Amer. Chem. Soc.*, 1894, xvi., No. 10, pp. 665-670.)—To prepare the fatty acids, 120 grammes of the fat are mixed with 45 c.c. of a solution of KOH (1,250 grammes per litre) and

* *Zeit. für Analyt. Chemie*, 30-738.

stirred at a temperature but slightly above its melting-point until emulsified. The beaker is then covered and kept at 100° until the fat is completely saponified. The soap is decomposed with 165 c.c. of dilute H_2SO_4 (specific gravity 1.143), preferably in a silver dish, and the fatty acids are washed, firstly with dilute H_2SO_4 (5 per cent.), and afterwards with water till the washings no longer taste acid. They are then dried in an open dish for two hours at 100°.

In the determination of the freezing-point, a thin-walled test-tube, 3.5 c.m. by 15 c.m. is fitted by means of a cork into a suitable bottle. A centigrade thermometer, graduated in fifths of a degree from 1° to 60°, is fixed into this tube by a cork loose enough to allow the contents of the tube to be stirred. The tube is filled with the melted acids to within about 1 c.m. from the top, and these are slowly stirred until partial solidification sets in, when the thermometer is rapidly turned round ten times and then allowed to stand, care being taken that it clears the bottom of the tube by 4 or 5 c.m. The highest temperature to which the mercury rises is taken as the freezing-point. Duplicate determinations should not differ more than 0.1° C.

• Experiments which led to the adoption of this method showed that the method of saponifying did not affect the melting-point, but that when alcoholic saponification was used, boiling the soap solution one and a half hours was necessary to completely remove the alcohol. The length of time in saponifying did not influence the final result.

It is essential that the fatty acids should be well dried, water lowering the freezing-point and concordant results not being obtained. Two determinations, using the same undried acids, gave melting-points of 43.14° and 42.86°. It is immaterial whether the dried acid be used immediately for the determination or be allowed to solidify and subsequently remelted.

The following determinations of the melting-point of the same fatty acids, using tubes of different sizes, prove that the test-tube may exceed 3.5 c.m. in diameter, but must not be less, otherwise the ratio between heat radiating from the fatty acid through the walls of the tube and the amount of liberated latent heat is disturbed :

		Test-tube 3.5 c.m. diameter.		Test-tube 2.5 c.m. diameter.		Difference.
		...	...	...	...	
Fatty Acid	1.	...	43.52°	...	43.34°	0.18
	2.	...	42.88°	...	42.65°	0.23
		Test-tube 3.5 c.m. diameter.		Test-tube 7 c.m. diameter.		Difference.
		...	...	...	...	
Fatty Acids		...	43.45°	...	43.46°	0.01

The error due to incomplete immersion of the thermometer may be almost eliminated by using a thermometer shortened by having an enlargement blown in the bore in the interval between 2° and 28°, thus diminishing the amount of mercury above the surface of the fatty acid.

C. A. M.

Determination of Volatile and Insoluble Fatty Acids in Butter Fat. W. H. Beal. (*Journal Amer. Chem. Soc.*, 1894, xvi., No. 10, pp. 673-676).—The process consists in decomposing the saponified fat with a 20 per cent. solution of ortho-

phosphoric acid, and expelling the volatile acids by means of a current of steam, the operation being usually complete when 500 c.c. have distilled over. The insoluble acids are washed until free from phosphoric acid, and dried on water-bath at 100° until they begin to increase in weight. Very uniform and concordant results are said to be obtained. The author disclaims previous knowledge of Goldman's method of distilling the volatile acids in a current of steam.*

C. A. M.

The Influence of Alum, Aluminium Hydroxide, and Aluminium Phosphate on the Digestibility of Bread. W. D. Bigelow and C. C. Hamilton. (*Journal Amer. Chem. Soc.*, xvi., No. 9, pp. 587-597.)—The authors have studied the question with especial reference to the double digestion of the gastric and pancreatic ferments. Their general method is the determination by Kjeldahl's process of the quantity of albuminoids in the material left undissolved after treatment with artificial pepsin and pancreas solutions.

Five processes are used :

1. *Digestion in Pepsin Solution.*—1 gramme Merck's granulated pepsin dissolved in 1,000 c.c. of 0·33 per cent. HCl, and 2 grammes of dried bread previously extracted with ether placed in flask with 100 c.c. of the acid pepsin solution. This is kept at 40° on water-bath for twelve hours, with frequent shaking. The contents are then filtered, and the residue washed, dried, and Kjeldahled.

2. *Stutzer's Method.*—2 grammes of bread are digested in the pepsin solution, and afterwards for six hours in Stutzer's pancreas solution.

3. *Stutzer's Method modified by Wilson.*—After treatment with the pepsin solution, the residue is digested for twelve hours at 40° in pancreas solution, made by dissolving 1·5 grammes Merck's pancreatin and 3 grammes sodium carbonate in 1 litre of water.

4. *Niebling's Method.*—2 grammes of the bread washed with ether and placed in flask with 100 c.c. of 2/10 per cent. HCl. Boiled for fifteen minutes, cooled, neutralized, or made slightly alkaline with sodium carbonate solution. 100 c.c. of Stutzer's pancreas solution then added, and the flask immersed for six hours in water-bath at 37° to 40°.

5. *Niebling's Method modified.*—The same as the preceding, except that the pancreatin solution given under Wilson's modification of Stutzer's process is used instead of Stutzer's pancreas solution.

The results of digesting bread free from alum are compared with those from bread containing known quantities of alum, aluminium hydroxide, and aluminium phosphate :

I. *Bread Free from Alum.*—Loaves made from flour known to be pure were cut in slices, dried at 98°, ground and bottled. They contained 12·06 per cent. albuminoids. The following percentages of the albuminoids were found to have been digested by the different methods :

Pepsin Solution.		Stutzer's Method.		Stutzer's Method modified by Wilson.		Niebling's Method.		Niebling's Method modified.
93·26	...	93·57	...	93·21	...	93·38	...	93·28

* *Analyst*, September, 1892, 174.

II. *Alumed Bread*.—Two loaves made from alumed flour. No. 1 contained 0·8 grammes of alum and 11·88 per cent. of albuminoids. No. 2 contained 4·28 grammes of crystallized alum and 12·06 per cent. albuminoids. Percentages of albuminoids digested :

	Pepsin Solution.		Stutzer's Method.		Stutzer's Modified.		Niebling's Method.		Niebling's Modified.
1.	89·11	...	92·56	...	92·21	...	92·54	...	92·74
2.	80·98	...	92·4	...	92·44	...	—	...	92·62

From these results the authors conclude that the influence of alumed flour on the digestion is over-estimated, since the albuminoids not digested by the pepsin are almost all digested by the alkaline pancreas solution.

III. *Bread containing Aluminium Hydroxide*.—Loaf No. 1 contained ·54 gramme ; No. 2, 2·5 grammes. Percentages of albuminoids digested :

	Pepsin.		Stutzer's Method.		Stutzer's Modified.		Niebling's Method.		Niebling's Modified.
1.	87·03	...	92·18	...	92	...	91·77	...	91·90
2.	86·78	...	90·43	...	90·21	...	89·13	...	88·96

IV. *Bread containing Aluminium Phosphate*.—Loaf No. 1 contained ·64 grammes ; No. 2 contained 3·2 grammes. Percentages of albuminoids digested :

	Pepsin.		Stutzer's Method.		Stutzer's Modified.		Niebling's Method.		Niebling's Modified.
1.	80·87	...	83·11	...	82·56	...	86·35	...	86·46
2.	71·21	...	78·26	...	81·32	...	82·18	...	81·74

The influence of aluminium hydroxide on digestion is thus shown to be about the same as that of an equivalent quantity of alum. The action of the phosphate is quite different, for, in spite of frequent statements as to its insolubility, the preceding results show that from 10 to 12 per cent. of albuminoids which are digestible in presence of aluminium hydroxide and alum are insoluble in the presence of an equivalent quantity of aluminium phosphate.

C. A. M.

Separation of Tin and Antimony in Alloys. Mengin. (*Comp. Rend.*, 1894, cxix. 224 ; through *Chem. Zeit.*).—Tin and antimony are first separated from the other constituents of the alloy by treatment with nitric acid. The mixed oxides are weighed and treated with hydrochloric acid and a rod of tin. On heating, the antimony is precipitated as metal, and the tin goes into solution as stannous chloride. The metallic antimony is washed with boiling water, dried, and weighed, the tin being obtained by deducting this value after calculation into oxide from the weight of the mixed oxides of tin and antimony originally obtained.

B. B.

NOTE BY ABTRACTOR.—The method in its present form is vitiated by the facts (1) that antimony is not completely separated as an insoluble oxide on treating an alloy containing it with nitric acid ; and (2) that metals other than antimony and tin are invariably retained by the oxides left on dissolving an alloy of this class in nitric acid. Accurate separation of the antimony and tin from the remaining metals of the alloy is, therefore, requisite before using the reduction process described above.

B. B.

Distinctions between Atropine and Strychnine by means of Vitali's Reaction. D. Vitali. (*Boll. Chim. farmac.*, 1894, xxiii. 449; through *Chem. Zeit.*).—When strychnine is oxidized with nitric acid, and the dry residue treated with alcoholic potash, it gives a red colour, which may possibly be confused with the similar reaction for atropine, which has been described by the author. The following distinctions exist, however: (1) On oxidation with nitric acid, especially immediately after the evaporation of the acid, atropine gives an agreeable odour, while strychnine under the same circumstances yields no smell. (2) Strychnine becomes yellow on oxidation, and the residue left on evaporation is also yellow; no such coloration occurs with atropine. (3) On evaporating the alcohol, after the addition of alcoholic potash, atropine gives a violet residue, which becomes more intense in colour on again treating it with alcoholic potash; with strychnine the residue is yellow or reddish-yellow, and becomes reddish-violet on repeated treatment with alcoholic potash. (4) When water is added after the colour has been produced by alcoholic potash, the coloration due to atropine is bleached, but that given by strychnine becomes yellow. (5) When atropine is oxidized with nitric acid, and the dry residue treated with ammonia, yellow drops are formed which become violet on the addition of alcoholic potash. Under like conditions strychnine gives with ammonia an orange-red, and when alcoholic potash is added, the violet colour produced is only transitory, becoming a strong blood-red. On shaking out the ammoniacal solution of the oxidation product with chloroform, and evaporating the solution in chloroform thus obtained, the residue given by atropine is nearly colourless, and takes a permanent violet colour when treated with alcoholic potash. Strychnine, on the other hand, when submitted to the same process, colours the chloroform slightly yellow, and yields on evaporation a yellow residue, which gives a strong orange-red coloration on addition of alcoholic potash.

B. B.

Contributions to Volumetric Analysis. B. Reinitzer. (*Zeitschrift für angewandte Chemie*, 1894, pp. 547-551, and pp. 573-579.)

Indicators.

In the opinion of the author litmus solution is the most serviceable indicator, excelling methyl orange in sharpness of change of colour and sensitiveness (about eight times as great), while it possesses an advantage over phenol-phthalein in being capable of being used in the presence of ammonium salts.

The disadvantage in its use is its great sensitiveness to carbonic acid, and special precautions are accordingly necessary in its preparation and use to avoid this source of error. It should be prepared from good litmus, poorer samples in the market being often almost useless, and since this contains alkaline carbonate, the solution must be boiled for seven or eight minutes and then neutralized with HCl, so that the wine-red colour remains even on further boiling. The solution is then cooled and an equal volume of strong alcohol added. The stock solution should be kept in a bottle with a delivery pipette inserted through the cork.

The final change of colour is sharpest when the liquid to be titrated is boiled for seven or eight minutes and then well cooled. In order to avoid the influence of atmospheric CO_2 it should not be allowed to stand exposed for long, and dilution with unboiled distilled water should be avoided. A similar source of error is that carbonate often occurs in standard alkaline solutions. The author found the same in calcium and barium hydrate solutions. In one litre of clear lime water, containing 1.1738 gramme of CaO , there was found .0093 and .0087 gramme of CO_2 . To avoid this it is necessary to so arrange that after boiling only a little alkali need be added.

Experimental proof of the inferiority of methyl orange as an indicator is given, showing that, owing to the fact that the final change of colour is not sharp but passes through intermediate changes, it cannot be used for accurate work with decinormal solutions, nor when the liquid to be titrated is of large volume (500-1,000 c.c.). In the former case, in titrating 250 c.c., twenty to thirty drops of decinormal acid are required to change the colour from clear yellow to the clear "onion" red; in the latter, even with only 100 c.c., six drops of decinormal acid = 0.19 c.c., are required for the change. On the other hand one drop decinormal alkali or acid is sufficient to change the colour of 250 c.c. of a neutral solution when litmus is the indicator.

Emphasis is laid on the point that the fluid must be cold when titrated, whatever indicator be used. Thus, a solution, which when cold only requires two drops = 0.06 c.c. of decinormal acid to change the colour of the litmus, requires five drops = 0.15 c.c. when heated to 100° . Similarly, phenol-phthalein is three times more sensitive in a cold solution than in a hot one. In the case of methyl orange a solution of 250 c.c., which shows a change of colour with sixteen drops of decinormal HCl . = .48 c.c. in the cold, requires 2 c.c. of the acid when heated.

It is suggested, as a possible explanation of this, that with the rise of temperature an increasing dissociation of the combination between the indicator and the acid or alkali occurs, so that the excess of either necessary to produce the colour also rises with the temperature.

Preparation and Use of Pure Na_2CO_3 as a Standard for Acids and Alkalies.

Pure Na_2CO_3 may be obtained by the following method: As much sodium bicarbonate as possible is dissolved in 250 c.c. of water at 80°C . and freed from insoluble impurities by filtration. On cooling down to a temperature of from 10° to 15° in a stream of water a double salt which has the composition of $\text{Na}_2\text{CO}_3 + \text{NaHCO}_3 + 2\text{H}_2\text{O}$ crystallizes out. Most of the soluble impurities remain in the mother liquor, which is sucked off, and the salt is further purified by repeated washing with small quantities of cold water. When heated in a platinum basin at a temperature just below perceptible redness, the residue is perfectly pure Na_2CO_3 .

The salt thus prepared should always be heated again immediately before use and the weighed quantity dissolved in warm water. In accurate determinations the temperature of the solutions must be taken into account: a liquid which measures 50 c.c. at 17°C . becomes 50.05 c.c. at 22° , which causes an error of 0.1 per cent.

Ammonium Chloride as a Standard for Acids, Alkalies, and Chloride Solution.

The salt is easily obtained pure, either by careful selection from the commercial salt, or by neutralizing ammonia solution with HCl. Its purity is tested in solution by ammonium sulphide, and by sublimation. When using it for standardizing acids the author employs a modification of the ordinary method of estimating ammonium in its salts. About 2.6 grammes are placed in the distilling flask, which is then about half filled with water. A stick of potash weighing about 10 grammes is introduced, and the flask heated with a Bunsen. The NH_3 liberated is conducted by a tube delivering downwards into a flask containing the acid to be standardized. About fifteen minutes is sufficient for all the gas to pass over. The receiving flask is boiled for six or eight minutes to get rid of CO_2 , cooled, and the unneutralized acid titrated back with a standard alkali.

As one example of the accuracy of the method the following may be quoted: 2.68692 grammes NH_4Cl by calculation correspond to 50.336 c.c. normal acid, while the amounts experimentally found were 49.71 and 49.74 c.c.

Too large an excess of potash in the distilling flask is to be avoided, since it increases the difficulty of expelling the ammonia.

In standardizing alkalies with ammonium chloride, the salt is placed in a flask of Schott's glass, 300 c.c. of water added, and an excess of the alkali. The flask is boiled until the escaping steam no longer smells of ammonia (fifteen to twenty minutes). Acid is run in to acid reaction and the flask boiled, cooled, and titrated back. The results tally with those obtained by the distillation method described above.

Influence of Boiling on Glass Vessels.

That a boiling fluid dissolves alkali in glass is shown by the following experiment:

To 300 c.c. of distilled water, placed in an ordinary boiling-flask, lime-water was added, and after boiling and cooling titrated with decinormal HCl. On boiling for fifteen minutes .03 c.c. additional acid was required, and the same quantity on again boiling for fifteen minutes.

With larger quantities of liquid the error caused by boiling for fifteen minutes was so increased that the advantage of using a decinormal acid was quite lost. 700 c.c. of distilled water boiled for four hours in a new Erlenmeyer flask required 9.53 c.c. of $\frac{1}{10}$ N acid to neutralize the dissolved alkali. After continuing the boiling for fifteen minutes the reading was 10.22 c.c., rising to 10.92 c.c. when again boiled for the same length of time.

A Bohemian hard-melting potash glass offered about ten times the resistance of ordinary glass. After four hours' boiling 700 c.c. required 1.09 c.c. $\frac{1}{10}$ N acid to neutralize the dissolved alkali, rising after ten minutes' longer boiling to 1.14 c.c., and to 1.16 c.c. after a subsequent seven minutes. Thus the amount of alkali dissolved by boiling liquids for not more than ten minutes in vessels of this kind of glass affects the accuracy of the result but slightly.

It was not, however, until he used vessels made of Dr. Schott's new Jena glass that the author was able to obtain perfectly satisfactory results in standardizing

solutions. In a flask of this glass 700 c.c. of distilled water boiled for four hours used 0.13 c.c. $\frac{1}{10}$ N acid, which after eighteen minutes' further boiling became 0.18 c.c.

C. A. M.

The Detection of Chlorine in the Presence of Bromine and Iodine.
A. Nieliers and M. Fayolle. (*Comptes Rendus*, 1894, cxviii., 1152 and 1204, through *Chem. Zeit.*)—The authors have devised a method for the detection of traces of chlorine, in presence of much bromine and iodine, which depends upon the relations of the three halogens to an acid solution of aniline. In such a solution iodine produces no apparent change, bromine causes the formation of a white precipitate, while chlorine gives a colored product varying according to its quantity from a definite black precipitate to a blue coloration. The delicacy of the reaction depends on the acidity of the aniline solution. The best results are obtained by the use of a mixture of 400 c.c. of a saturated aqueous solution of aniline and 100 c.c. of glacial acetic acid. This mixture can be kept in yellow bottles, and then remains free from colour. The process of analysis is as follows: 10 c.c. of the solution to be examined are placed in a flask, and 5 c.c. of a mixture of equal parts of sulphuric acid and water, and 10 c.c. of a saturated solution of potassium permanganate are added. The flask is gently warmed, and the vapour therefrom caught in a well-cooled receiver containing some of the aniline solution specified above. If chlorine alone be present in the original solution, as little as 0.1 milligramme gives a perceptible blue coloration, which on warming quickly becomes red. Iodine is oxidized in the flask and does not distil. Bromine, however, comes over and forms a precipitate in the aniline solution, the precipitate being colored only if chlorine be also present. The presence of bromine interferes considerably with the delicacy of the reaction from the formation of bromine chloride. This drawback is avoided by the following preliminary treatment: The halogens in the solution to be tested are first precipitated in the ordinary way by silver nitrate, and the mixed halogen salts of silver thus obtained are washed and allowed to stand for some time under weak ammonia solution (1 c.c. of strong ammonia diluted with 10 c.c. of water). The silver chloride is dissolved, the bromide and iodide being but little affected. The solution is filtered, the ammonia evaporated, and the silver thrown down with sulphuretted hydrogen, and the solution filtered. The excess of sulphuretted hydrogen is then driven off by heating, and the solution concentrated to 10 c.c., and put through the process already described. It should be noted that a cork should not be used for the distilling-flask, but a ground-in stopper carrying a delivery-tube and a safety-funnel. By this modified method the smallest trace of chlorine can be detected in bromine and iodine compounds. In the case of the presence of cyanogen this substance is removed before the examination for halogens is begun. B. B.

EXTRACTS FROM THE EVIDENCE GIVEN BEFORE THE SELECT
COMMITTEE ON FOOD PRODUCTS ADULTERATION, ON
JULY 11, 18 AND 25.

MR. RICHARD BANNISTER.

(Continued from page 231.)

5. EVIDENCE REFERRING TO MARGARINE IN BUTTER.

607. With regard to butter, you have changes taking place in butter ordinarily when it is kept for any length of time, have you not, which makes some difficulty in analysis?—There is a very great difficulty in the analysis of butter.

608. In what way?—Because, keeping the butter, it gets rancid, and we find that the insoluble fatty acids increase rather largely and the soluble fatty acids diminish; and inasmuch as if the insoluble fatty acids are high, the probability is that foreign fats may have been added, it follows as a matter of course that if you get a fresh butter to which fatty acids within certain limits have been added, the result of the analysis may be the same as the examination of an old butter that is perfectly genuine.

609. That introduces confusion and causes a difficulty of detection?—Very great difficulty of detection.

610. Butter was, I think, the next most frequent article submitted to you after milk?—Yes.

611. And the chief difficulty that you find in the butter analysis is the detection of foreign fat?—Just so.

613. In what form is the foreign fat introduced?—It is rather difficult to say, because there are so many ways of introducing this foreign fat, but very often it is introduced in the form of *stearine* or margarine.

627. Where do the butters come from chiefly that are adulterated with foreign fats, such as margarine?—Some from France and some largely from Holland.

628. And from Belgium?—Chiefly from Holland.

666. Margarine, I think, in its purest form is practically refined beef-fat, is it not?—It is.

667. Is it, then, fat of the same nature which exists in milk?—*Yes*.

685. But in inferior kinds of margarine coarser oils are used, such as those of the coco-cotton plant and the palm, which are substituted for the finer oils?—And a large quantity of sesame oil is used too.

686. And these are mixtures that are sold in the market sometimes as butterine-porcine; have you heard of that, or lactine, or cocoa-butter, or lardine; have you heard these expressions used?—Yes, I have heard those expressions.

687. Those represent the inferior kinds of margarine?—*Yes*.

688. And those are either mixed with butter or churned with milk, and they make up some of the various compounds sold as margarine, as we have heard?—*Yes*.

1779. The Minister of Agriculture asked you the other day whether you had ever heard of an article called lactine, or cocoa-butter, or lardine, and as to whether these articles were inferior kinds of margarine. Do you consider that those articles enter into the composition of margarine at all?—*I do not know*.

1780. Is not this lactine refined cocoanut-oil?—It is.

And lardine is a lard substitute?—Yes.

1783. And not in any way whatever sold as a low character of margarine?—*It is not.* (See reply to question 687, page 259.)

959. What is arachis; what substance is it taken from?—*I do not know its origin.*

691. What percentage is the lowest that it is possible to detect of animal fat in butter?—It is a very difficult question to answer, because very frequently there are certain substances used with the animal fat for a particular purpose, and on account of the presence of those substances you may detect a very small percentage, when, if you depended entirely upon the animal fat you could not detect it; you could not say positively that it was there.

692. Will you explain that last answer a little more?—Certain of these vegetable oils that are used in the manufacture of margarine have distinct chemical reactions, and if they are present, of course, the presence of this oil assists you in the determination of the presence of fat.

693. You say that the presence of these oils indicates the presence of fat; but it could not enable you to estimate the percentage of margarine itself, could it?—Very frequently when examining a sample of butter that contains a certain percentage of foreign fat you are quite certain from the analysis that there is something wrong with the butter; that it is either an old butter or something of the kind; and then you may get these indications as you go on in the analysis of samples that determine you, before you come to the end of your examination, that margarine is really present in a sample of butter.

699. Mr. Herbert Gardner asked you about the use of preservatives in milk and butter, and you mentioned especially boracic acid; I suppose that is in the form of the boro-glycerine?—*It is generally used in that form.*

706. I ask you as a matter of fact, would you treat the presence of a little boracic acid, if you found it in butter, differently from the presence of some chloride of sodium?—I should not.

934. I want to ask you a few questions as to the test you apply (I do not think that you have given any evidence on that point) to determine the presence of margarine in alleged butter. Perhaps, as you have given the tests with regard to milk, it would be of value to the Committee if you could give briefly in clear popular terms what tests you consider satisfactory with reference to determining the presence of foreign fats and the proportion of foreign fats in any sample of butter?—Perhaps it would be better to start with the general principle first. In a butter we have there a certain percentage within certain limits of insoluble fatty acids, and a certain proportion within certain limits of soluble fatty acids. In a foreign fat nearly the whole is insoluble fatty acids. It follows that if a foreign fat be added to butter it will increase the percentage of insoluble fatty acids and diminish the quantity of soluble fatty acids. In the analysis of the sample we determine the quantity of the proportion of insoluble fatty acids, and also the proportion of soluble fatty acids. When we have these percentages we have to go back to the variations in genuine butters in the insoluble and soluble fatty acids; then from the physical conditions of the sample,

from the results of the analysis, we have to determine whether the sample that we examine is a genuine butter or whether it contains foreign fat.

935. Can you tell that with absolute accuracy?—You cannot, I am sorry to say, because the butter varies largely in composition itself.

936. I suppose that the fat in milk and the fat in the bullock would be the same?—To a certain extent—not entirely; you get a slightly different composition in butter from what you get in the fat of the bullock.

937. I think in your previous answers you said that margarine exists in milk?—Certainly.

938. The chemical fatty acids exists, do they not, in all these dairy products?—Yes; but when you come to ordinary margarine, in an ordinary butter, there is something else besides that; the fats of margarine do exist in butter, plus the soluble parts that do not exist in the margarine.

939. Do the insoluble fatty acids exist in the milk?—Yes.

941. Plus other substances?—Yes; plus other substances.

944. How do you determine the proportion of foreign fats present?—We can only do that when we get beyond certain limits.

945. What are these limits?—I will give you a case in point: If you take a fresh butter, the probability is that you will get of insoluble fatty acids about 87 or 88 per cent.; in the case of a fat, you will get of insoluble fatty acids something like 96 per cent.; and when you go on the basis of calculation of, we will say, 87 to 96, 9 per cent. represents all the difference that there is between the genuine butter on the one side, and margarine on the other side. Now, in the case of butter that has been kept you may have your insoluble fatty acids go up as high as 89 per cent.; it follows, then, as a matter of course, if you take your first 87 per cent., that you have got 2 per cent. there in the butter of change, and that may be either due to the change of decomposition in the butter itself, or it may be due to the addition of foreign fat; but the 2 divided by 9 shows a 20 per cent. margin for adulteration.

1757. So that it must follow that if you get a fresh butter to which margarine, in certain limits, has been added, the result will be the same as the examination of old butter that is genuine?—Yes.

1759. Of course the analyst would have to state on his certificate whether the butter, as in the case of milk, had undergone any changes that might interfere with the analysis?—Yes.

1760. And he would be in a preferable position for correct analysis, dealing, as he would deal, with perfectly fresh butter?—He would have the butter in a physical condition that would certainly be better than when we got it further on, if it had not been kept properly.

950. Are you satisfied with the test that is called by the other name of saponification?—*That is the test I have been describing.*

951. And which you adopt?—Yes.

952. Does not that work out in figures?—Yes; but only in the limits I tell you, because the butter itself may be so different in constitution owing to its physical condition that it gives so much more margin for the addition of foreign fat.

953. Does the condition make a very considerable difference in the amount of

potassium which is absorbed?—Yes, it does, because that has all to be worked out into percentages again.

954. I see that 2·27 milligrammes is used in America as about the standard of pure butter?—That may be taken as the standard of pure butter, but at the same time they take into account the alterations in the physical condition of the butter, and therefore they cannot make a sharp line.

955. You throw serious doubt on the wisdom of American analysts and other analysts, like Hehner, who have employed these tests, in selecting as the figure 2·27 milligrammes of potassium hydroxide. What is the test that you would apply?—I should like to look into the whole question if you will allow me. If you will ask me the question on Wednesday next I shall be happy to answer it.

(Note the confusion between the different methods of analysis, and the acknowledgment, on the part of the Referee under the Food Act, that he has to look the matter up before being able to reply.)

957. There is one question that I want to ask you with regard to these tests which I think you will be able to answer at once; that is, that those oils, to which Mr. Herbert Gardner drew your attention in his examination, will require a higher percentage of potassium than pure butter, will they not?—Yes.

(Note.—After looking the matter up, Mr. Bannister again refers to the subject as follows:)

1525. There was a question which you wished to consider further with regard to the testing of the fats by the saponification method, whatever that may be; I asked you a question as to the figures used by the American analysts, and especially as to the number of 227 milligrammes of potassium hydroxide to test the quantity that butter would absorb, as compared with other fats that absorb less; and I think you said that if I asked you the question to-day you would answer it?—I shall be very happy to do so. The 227 milligrammes there is mentioned in connection with a test called the Koettstorfer's test, and the number is 227; that refers to butter-fats. But when we come to look at the table itself we find that 227 is the average of the butter experiments; the minimum is 221·5, and the maximum 232·4. I think you will find that the average of those two figures is 226·9.

1526. May I ask you what range these experiments appear to have been taken over, a very large range, or over a limited range?—Over a very large range of different kinds of butter, and also of different kinds of fat. I thought you would like me to work out the result as given above, when I explained the difficulty that there was of determining a small quantity of foreign fat in butter. If we take these figures, the minimum figure of 221·5 and the maximum figure of 232·4, between them you would be able to put into the better description of butter about 30 per cent. of foreign fat to make it come up to the higher one.

1527. That amount could be let in without detection?—Without detection.

1528. Would it be of use if you were to state the average figures for the other fats?—*I can scarcely understand about the other fats; lard oils and the oils to which the test was applied.*

1529. The oil, I understand, takes up a larger quantity, but the organic fats which are used for adulteration take a less quantity, I understand?—Koettstorfer's

number for the butter-fat is 241 to 253, and in the case of arachis, or earthnut-oil, it is 285 to 296; but so far as this test is concerned, after looking it over very carefully, and trying it ourselves, and also after comparing the results of other examinations by other chemists, it is put on one side as not being a sharp test for the detection of foreign fat in butter. (Note the confusion between milligrammes of KHO and the saponification equivalent.)

1534. What is the sharpest test that you do apply?—I think that the sharpest test of all is the estimation of the soluble acids in butter, that is to say, the acids soluble in water.

1535. Is that the test that you refer to when you said that you had to take the physical condition of the butter into consideration, as well as the results of the analysis?—THAT IS SO.

6. EVIDENCE REFERRING TO THE AMOUNT OF WATER IN BUTTER.

615 and 617. During the last two or three years you have had a good many samples (of butter). Have you anything to say about the analysis of these samples?—A system of adulteration that seems to me to have come in lately is putting in too much water; that seems the modern way of adulteration; and there is no doubt that during the last few years there has been a considerable increase in the quantity of water present in butter, in particular kinds of butter.

619. The butters that contain an excess of water are very often Irish butters, are they not?—Yes, they are.

620. And this water has been pressed into them for the purpose of adding to the weight, I suppose?—It has been worked in for the purpose of adding to the weight.

622. What is the standard of water in genuine butter?—There is no standard. In fresh butters it will go from about 12 to 14 per cent. But when we come to salt butter it will go up as high as 16 per cent. or a little more, but those samples contain sometimes as much as 24 per cent.

1770. The strong evidence given before the magistrates at Manchester on the part of Irishmen interested in butter was that it was perfectly consistent with the butter being legitimate that it should contain 20 per cent. of water?—There is no necessity for it to contain 20 per cent. of water.

1776. I have had brought to my notice a certificate given by Dr. Bell in his book, where he included under the head of genuine butter a sample containing as much as 20·75 per cent. of water?—That is a very long time ago, and that was a private sample.

1777. You would not pass a sample now with 20 per cent. of water?—Not without making a remark about it.

1778. You would not pass it as genuine, would you?—No.

2731. Are you aware that Somerset House has passed butter as pure which contained 19 per cent. of water?—No; I do not think we have done so.

2732. Are you aware that Dr. Bell stated that pure butter will sometimes contain over 20 per cent of water?—I think I can explain where we are differing. Those samples which you find in Dr. Bell's book were samples that we had obtained in order to see the quantity of water in commercial butter at that particular time, and

there is no doubt that the sample of butter there mentioned was a pure butter so far as its manufacture was concerned, and that it did contain that quantity of water; but it was not a reference sample.

2733. But it was a pure butter?—Yes.

(*To be continued.*)

REVIEW.

FERTILIZERS AND FEEDING STUFFS. By BERNARD DYER, D.Sc. (Lond.). London: Crosby, Lockwood and Son, 1894. Price 1s.

The little work before us consists of a series of articles from Dr. Dyer's pen which have appeared from time to time in various newspapers, and are now collected and reproduced in book form. To these an appendix is added which contains the text of the Feeding Stuffs Act of 1893 (annotated by A. J. David, B.A., LL.M., of the Inner Temple), and the forms and regulations of the Board of Agriculture. The first chapter is devoted to the consideration of the general functions of fertilizers. The second and third treat respectively on farmyard manure and artificial fertilizers. Chapter iv. deals with the application of artificial fertilizers, and chapter v. treats on purchased feeding stuffs. In chapter vi. the comparative values of feeding stuffs are commented upon. The book, as stated in the preface, is addressed to practical farmers, and not to agricultural students, consequently its contents are couched in plain, homely and familiar language; nevertheless, the information given is solid, sound, and thoroughly reliable. The appearance of the work is extremely opportune, for evidently it is just for want of such information as is here supplied that the Feeding Stuffs Act has been so far practically a dead letter. Any individual of ordinary intelligence cannot fail, after a careful perusal of the work, to be able to form a fairly competent opinion on the value of a fertilizer or feeding stuff from the results indicated by its analysis. The book also abounds in practical hints, not only derived from the subject as considered from a scientific point of view, but also from the author's extensive experience in actual farming operations. It cannot fail to be of the utmost value to those to whom it is specially addressed, whilst the agricultural student, to whom it is not addressed, may peruse it with advantage.

W. J. S.

CORRESPONDENCE.

To the Editors of THE ANALYST.

County Analyst's Office,

Darlington, October 10th, 1894.

SIRS,—The abstract in the current number of THE ANALYST which deals with a "Comparison of the Kjeldahl-Wilfarth and Stock Methods of determining Nitrogen," by E. Cavazzani and A. Cecconi, has occasioned me a little surprise. My belief is that these gentlemen have been misled by some error in translation, because in working my method I allow the sulphuric acid to act for some time before adding MnO_2 , and therefore the question of frothing is new to me.

I should not have intruded upon your space with respect to the conclusions at which Messrs. Cavazzani and Cecconi have arrived, if it had not happened that for two years I have used my process exclusively as Analyst to four important Agricultural Societies whose guarantees are of the strictest; and I have never had a single dispute, although my work has been checked by at least half a dozen different analysts acting for the vendors of both fertilizers and feeding stuffs. I venture to think a test like this is a little more to the point than a few experiments made in connection with an evidently imperfect knowledge of the method.—Yours obediently,

W. J. KEATING STOCK.